N° III.

IMPROVED APPARATUS FOR THE ANALYSIS OF ORGANIC PRODUCTS.

The LARGE SILVER MEDAL *was this Session presented to Mr.* J. T. COOPER, *of Paradise-street, Lambeth, for* AN IMPROVED APPARATUS FOR THE ANALYSIS OF ORGANIC PRODUCTS. *The following Communication has been received from him on the subject, and the Apparatus has been placed in the Society's Repository.*

AN easy and accurate method of determining the ultimate elements of bodies composed of carbon, hydrogen, oxygen, and azote, has been of late years a great desideratum among chemists, as such a variety of contrivances have been suggested by scientific individuals, all of which have their peculiar merits and defects. It is presumed the instrument and method of operating now presented to the Society and the public, if not entirely, may be considered as nearly free from those objections which in my opinion may be fairly urged against those heretofore in use. It might, however, be considered ungenerous, was I to take upon me the task of pointing out those defects, I shall therefore content myself by briefly stating in this communication, the class of substances to which it is applicable with a view to determining the pro-

portions of their elements, and a description of the method of operating upon each of them.

As this apparatus seems more particularly calculated than any other for operating on volatile matter, such as the essential oils, camphor, benzoic acid, and a variety of similar substances, I shall in the first place describe the method I have adopted in the analysis of this class of bodies; and when it is considered that I write not for those who are accustomed to the more minute and delicate operations of chemical analysis, but for those who are or may be considered as unacquainted for the most part with this subject, I hope I may not be considered as tedious should I venture to give those directions which to the more matured in science may seem to be unnecessary.

The oxid of copper used in the experiments is best procured from the residuum of verdigris (binacetate of copper), which is or was used to be distilled in glass retorts for the preparation of strong acetic acid. The reason I prefer the oxid of copper prepared by this process over any other is, that it is more likely to be free from impurity than that which is prepared by precipitation from acid solutions. Every one who is in the habit of preparing precipitates, knows the difficulty there generally is in freeing considerable quantities of precipitated matter from adhering neutral salts; and as the smallest impurity would in some measure contaminate the result of the analysis, it is a very necessary precaution that the oxid, which is by far the greatest in quantity of any substance that is employed in the operation, should be perfectly pure. Should it however happen that at any time such an oxid is not readily to be procured, the oxid that is obtained by heating copper plate and quenching it in water may be substituted; although I give the decided preference to the former on ac-

count of its mechanical texture being much more porous, and consequently exposing a larger surface to the action of substances in vapour passing through it, neither is it so likely to choak up the tube and endanger its bursting, and of course a failure in the experiment. Supposing the residuum above mentioned to be employed, it is requisite to expose it to a red heat for twenty minutes or half an hour to destroy the carbonaceous matter that invariably accompanies it; it should then be pulverised and sifted through a fine wire sieve; that portion which has passed the sieve being again sifted through a fine cyprus or lawn sieve, the finer dust is got rid of, and each of these portions may be separately kept, and is applicable to different purposes.

A tube of hard glass, either of crown or green bottle glass, being selected about fourteen or fifteen inches long, and from one to two tenths of an inch internal diameter, clean the inside from dust by passing through it a piece of cotton, then make it as hot from end to end as the fingers can conveniently bear, and draw air through it into the mouth (but not blow through it) while it is still hot, to ensure its perfect freedom from adhering moisture on its inside, and while still warm seal up one end with the blowpipe; the tube may be now balanced, but it is necessary in this, as in all other operations of analysis where very small quantities are concerned, that the beam should be affected by $\frac{1}{200}$ or $\frac{1}{300}$ of a grain, even when loaded with 4 or 5 hundred grains at each end.* The substance intended for analysis is now to be introduced into the tube, if it be solid, as for instance camphor or a like substance, it may be broken

* The balance I have been in the habit of using was made for me by Mr. Robinson, and is sensibly affected by $\frac{1}{1000}$ of a grain when loaded with 1000 grains at each end.

into small fragments and shaken down to the bottom; if it be a fluid, as a volatile or fixed oil, it may be introduced by means of a small funnel, as is shown in fig. 7, which funnel is prepared, on the instant, from a piece of flint glass tube of convenient size and substance, by heating it near one of its extremities, and suddenly drawing it out, it is evident the semifluid glass will be thus elongated, and a funnel with nearly a capillary tube and of any required length, may be thus obtained; a very little practice will render this part of the business very easy to be accomplished; the funnel is to be put into the tube, reaching very near its bottom or sealed end, and the fluid matter introduced without soiling the upper part of it; care must also be taken on withdrawing the funnel, that no portion of the fluid is attached to its lower extremity, or otherwise this will happen. The volatile substance, or that which is capable of being rendered so by a red heat, being now introduced into the tube, its weight is to be very carefully taken, which when done the oxid of copper previously freed from its fine dust by the lawn or cyprus sieve* and recently heated red hot, is to be poured into the tube while warm, to the length of 8 or 10 inches, having previously put into the tube as much only of perfectly cold oxid as will absorb the fluid portion of matter, and about a quarter of an inch above it, or to stand about the same height above the solid substance. Why I recommend the present proceeding is, that a small quantity of the cold oxid only is used to prevent the hot oxid from coming in contact with the volatile matter which might otherwise endanger the escape of a small portion from the tube, and of course would give erroneous results; and that portion of

* The finer portion is taken from the oxid to allow more freedom of passage for the vapour through it, in some cases the rush of gas is so sudden, was it not for this precaution, it would be likely to burst the tube.

cold oxid, even if it be fully saturated with moisture, can contain such a very minute quantity of water as not to sensibly affect the accuracy of the analysis. Having proceeded thus far, a quantity of recently-ignited asbestos, or spun glass (the former is best), is put into the tube, so as to occupy an inch or two, depending on the quantity of water that is expected to be formed; this must not be crammed but put rather lightly into the tube. The tube is now to be bent as represented in fig. 1, and its weight may be again taken, but this is not absolutely requisite, it is however well to do it. The tube is then to be covered with thin sheet copper, and placed between the forceps as represented in the same figure, with its open extremity inserted under a jar in the ordinary mercurial pneumatic trough, or it may be connected with a gasometer of Mr. Pepys's construction, which when ten or twenty grains of a substance are employed, and the quantity of either carbon or azote it contains is considerable, is convenient. Small mercurial graduated jars may be used, even if very large quantities of gas are obtained, as the process of decomposition may at any time be stopped almost instantaneously,* and the quantity contained in them being registered, they may be alternately filled with mercury and displaced by the gaseous products, as long as any comes over, reserving only the last portions for examination, of which a few cubic inches alone are requisite.

The lamps being trimmed with very short wicks are now to be lighted, lighting those first that are nearest the gasometer, and when the tube is red hot the remaining ones may be set fire to in succession, until the whole length of tube that is filled with the oxid is red hot. One set of lamps for a tube of the size I have mentioned above is generally sufficient, but

* I consider this as one of the advantages of this apparatus.

should tubes be used of larger size, such as half an inch in diameter, both sets will then be required. In coating the tube with sheet copper care must be taken not to cover that part of it which contains the asbestos, otherwise the heat will be conducted by it to that portion of tube and prevent the condensation of the vapour of water, which is very essential; and in placing the tube between the forceps, it will be convenient to allow that part of it which contains the volatile matter to project beyond the forceps, the heat that is conducted by the copper coating is generally enough to volatilize most substances. In the analysis of substances containing much hydrogen, and especially when ten or twelve grains of them are taken, it will be found convenient to attach to the tube a small bulb to contain the water that is generated; this is represented by fig. 6. I believe I have stated the whole that is necessary as respects the management and use of this apparatus as far as regards the decomposition of volatile substances; in the next place, I shall speak of its application to the decomposition of fixed substances, which after what has been said will require but very few words.

If the substance be a vegetable salt it must be freed from all extraneous moisture, this is best effected by suffering it to remain over an hygrometric substance *in vacuo* for some time.

Those who have not the convenience of an air-pump, may content themselves, by operating in this way, which although not quite so elegant, answers the purpose extremely well. A wide-mouthed phial provided with an accurately-ground stopper being procured, select another and much smaller phial that will easily go into it, and allow the stopper of the larger one to close accurately; it is as well to apply a little tallow to the stopper to ensure its more perfect fitting; strew on the bottom of the larger phial a quantity of chloride of calcium (dry mu-

riate of lime), put into the smaller phial the substance in fine powder intended to be dried, and place this in the larger phial standing on the chloride; moisten a small piece of bibulous paper with alcohol, and put it into the larger phial, but not inside of the smaller one; when thus arranged set fire to the moistened paper, and when it has burned a second or two put the stopper in its place; a very good vacuum is by this means formed, and the process of dessication goes on rapidly. I have repeatedly used this method and found it succeed very well; I think equally so with that usually adopted by means of the air-pump; although by some it may be ridiculed in these days of elegance and refinement.

The substance in this state is to be mixed with a portion of the oxid recently ignited, but in this case suffered to cool then as quickly as possible introduced into the tube. As much of the oxid may be used as would occupy an extent of tube equal in proportion to that shown in fig. 5; a quantity of oxid is then to be put upon the mixture, and over this it is sometimes well to put a small quantity of copper-filings or scrapings; upon these the asbestos is to be used as above, and the operation of ignition is to be conducted in a somewhat different manner to that last-mentioned.

The lamps in this case are to be lighted at the extremity next the gasometer, and as soon as the gas ceases to be liberated, the next in succession may be employed, and so on to the end; but instead of suffering the whole of them to continue in flame, it is as well to extinguish a portion, and to suffer only about three or four to remain in operation at once, but taking care to ignite the whole extent of tube at the close of the process. The gaseous products being collected, and their bulk noticed, their analysis is to be conducted in the usual manner, taking care, however, in all instances to observe the

precise temperature of the gases, that their bulk, as also the quantity of aqueous vapour they contain, may be estimated,* and either to equalize the internal and external surfaces of the mercury, or to calculate the volume of gas by the difference of mercurial levels.

Reference to the Drawing of Mr. J. T. COOPER'S *Apparatus for Analysis.—Plate II.*

Fig. 1, *a a* and *b b*, two long spirit lamps each having ten burners and wicks, the burners of each lamp sloping towards those of the other, as seen in the end view, fig. 2; they are placed in a tin tray, *c c*, mounted on four feet; this tray is perforated in the middle the whole length of the lamps, and as wide as *e e*, fig. 2; the object in making the burners sloping is that they may clear the lamps and approach each other as near as requisite, and yet leave a clear current of air to the flames, and the tray is perforated and mounted on feet to admit this current.

d d, are springing wires placed at each end of the tray to receive the tube *f f*, which contains the substance to be analysed, and to hold it over or between the two rows of flames; by pressing the finger and thumb on the two shoulders, *g g*, fig. 2, the wires open to receive the tube, and close on letting go; and should the tube be shorter than the lamps, an additional support on a leaden foot, fig. 3, is placed through the opening *e e* of the tray to rise between the flames, and hold the end of the tube; the tubes are hermetically sealed at one end, and the materials then put in while the tube is straight; it is then bent at the other end to suit the mercurial trough.

* For which very convenient formulæ will be found in the ninth edition of Dr. Henry's Elements of Chemistry.

The tubes are coated with copper foil, wrapped spirally round them; if each succeeding fold lie on half the other there will be a double coat of copper all the way; if it lie on two-thirds, there will be three layers of copper, and so on; by which the glass tube is supported from bending when hot, and becomes very uniformly heated. The spirals are continued beyond the end of the tube, to reach the support, and leave the end within the flames. The dotted lines at *h*, fig. 4, shew the end of the tube, short of the support; the foil is secured at the last coil by binding wire, as at *i*.

Fig. 5 shows the foil in act of being wrapped on, also the proportion of the space occupied by the materials; first, the mixture of oxide of copper with the material to be analysed, next pure oxide of copper, or copper filings, and, lastly, asbestos. When the quantity of water formed is considerable, the tube is either blown into a bulb, as at *k*, fig. 6, or melted on to one ready prepared as at *l*.

Fig. 7 is a long funnel, made by drawing out the end of a tube of a suitable thickness at *m*, till it is long and small enough through *n n* to reach to the bottom of the tube, and then cutting it off at *m*, by which liquids may be introduced to the bottom of the tube without wetting the sides.

As the wicks nearest the trough are to be lit first and the remainder in succession, as the former finish their action, there are upright supports of tin, *o o*, fixed on the lamps, one for each space between the burners, against which to rest a slip of tin *p p*, to prevent the lighted wicks from kindling those next, and it also enables the experimenter to blow out those that have done their duty. In fig. 2, the tin slip, *p p*, is shown by dotted lines, reaching from lamp to lamp: little flat caps are put on each burner when done with, to prevent the waste of spirit. Fig.

8 shows one of these caps, *q*, on its place; *r r*, fig. 1, a shelf fixed to the mercurial trough, to hold the lamps; *s s* the graduated jar. Tin pipes with corks, *w w*, as shown in fig. 2, are the apertures to pour the spirit into the lamps; their places only are marked at *w w*, fig. 1.

N° IV.

HYDROMETER FOR SALINE SOLUTIONS.

The GOLD VULCAN MEDAL *was this Session presented to Mr.* J. T. COOPER, *of Paradise Street, Lambeth, for a* HYDROMETER FOR SALINE SOLUTIONS. *The following Communication has been received from Mr. C. on the Subject, and a Set of the Apparatus has been placed in the Society's Repository.*

THE inconvenience and inaccuracy attending the hydrometers that have hitherto been in use for determining the specific gravity of acids, acid and alkaline solutions, solutions of neutral salts, &c. has caused me to direct my attention to the construction of an instrument that should be free from those objections; accordingly, it is presumed the instrument now presented to the public through the medium of the Society's transactions will be found convenient in its application, and accurate in its results.

In the present communication I shall venture to give those